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[54] **FLASK SEALING SYSTEM AND MICROWAVE TREATMENT APPARATUS WITH SUCH A SEALING SYSTEM**

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[52] **U.S. Cl.** **73/863.11**; 220/240; 219/756

[58] **Field of Search** 219/679, 687, 219/688, 704, 707, 756, 734, 772, 385, 428; 220/203.01, 203.08, 240, 315, 323, 324, 326; 73/863.11

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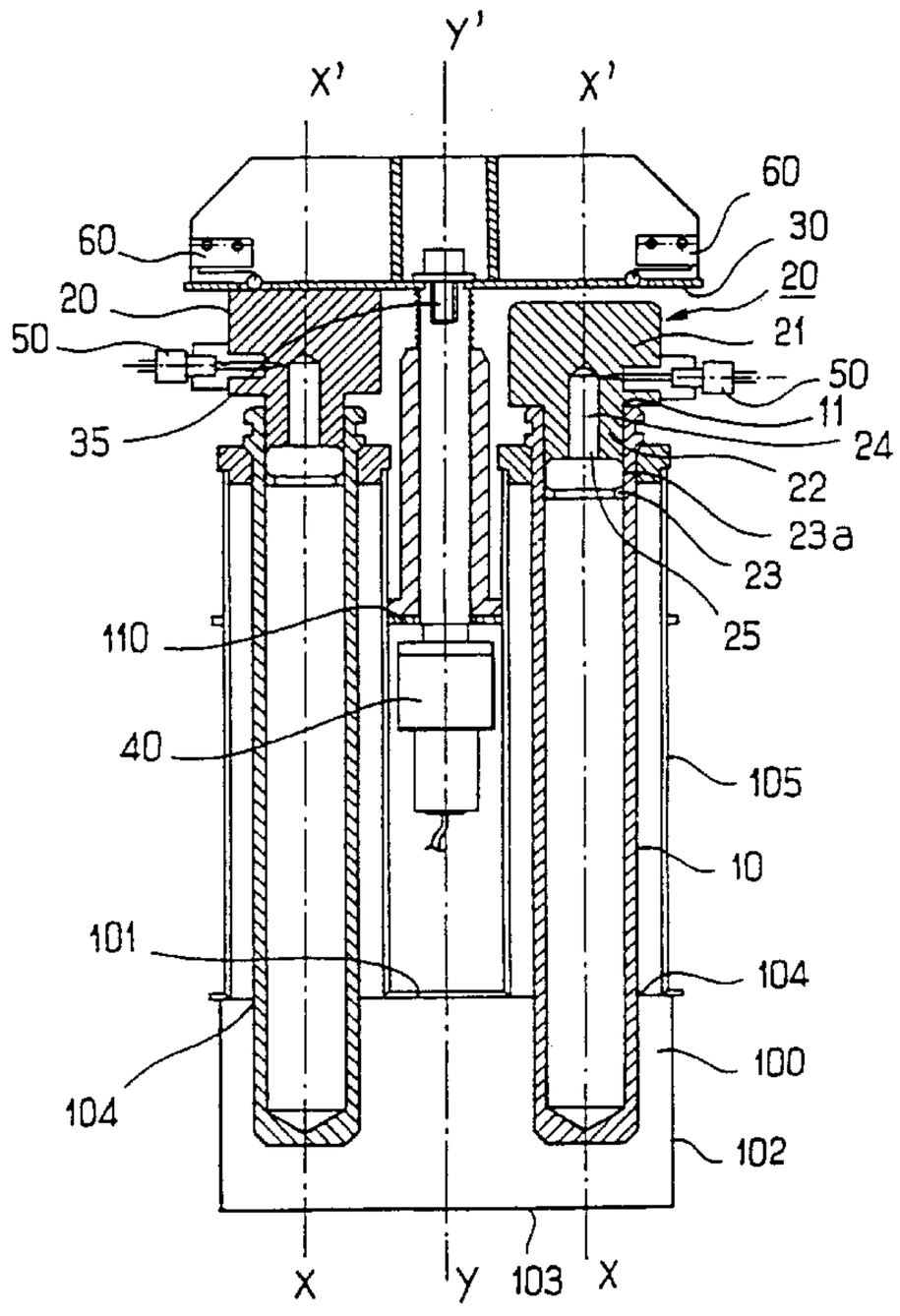
[57] **ABSTRACT**

A sealing system for sealing one or more flasks **(10)** with a longitudinal X–X' axis, open at a first end thereof **(11)** for introducing a sample and positioned inside microwave apl-lication cavity **(100)** for heating the sample is described. The system of the invention comprises a cap **(20)** to be partially inserted in the flaask along the X–X' axis so as to close the open end thereof and retaining meaans **(30)** for holding the cap in a flask sealing position, wherein the portion **(22)** of said cap which is inserted in the flask has a peripheral skirt **(23)** capable of sealingly engaging the inner wall **(12)** of the flask under an excess pressure created therein by heating the sample with microwaves.

13 Claims, 2 Drawing Sheets



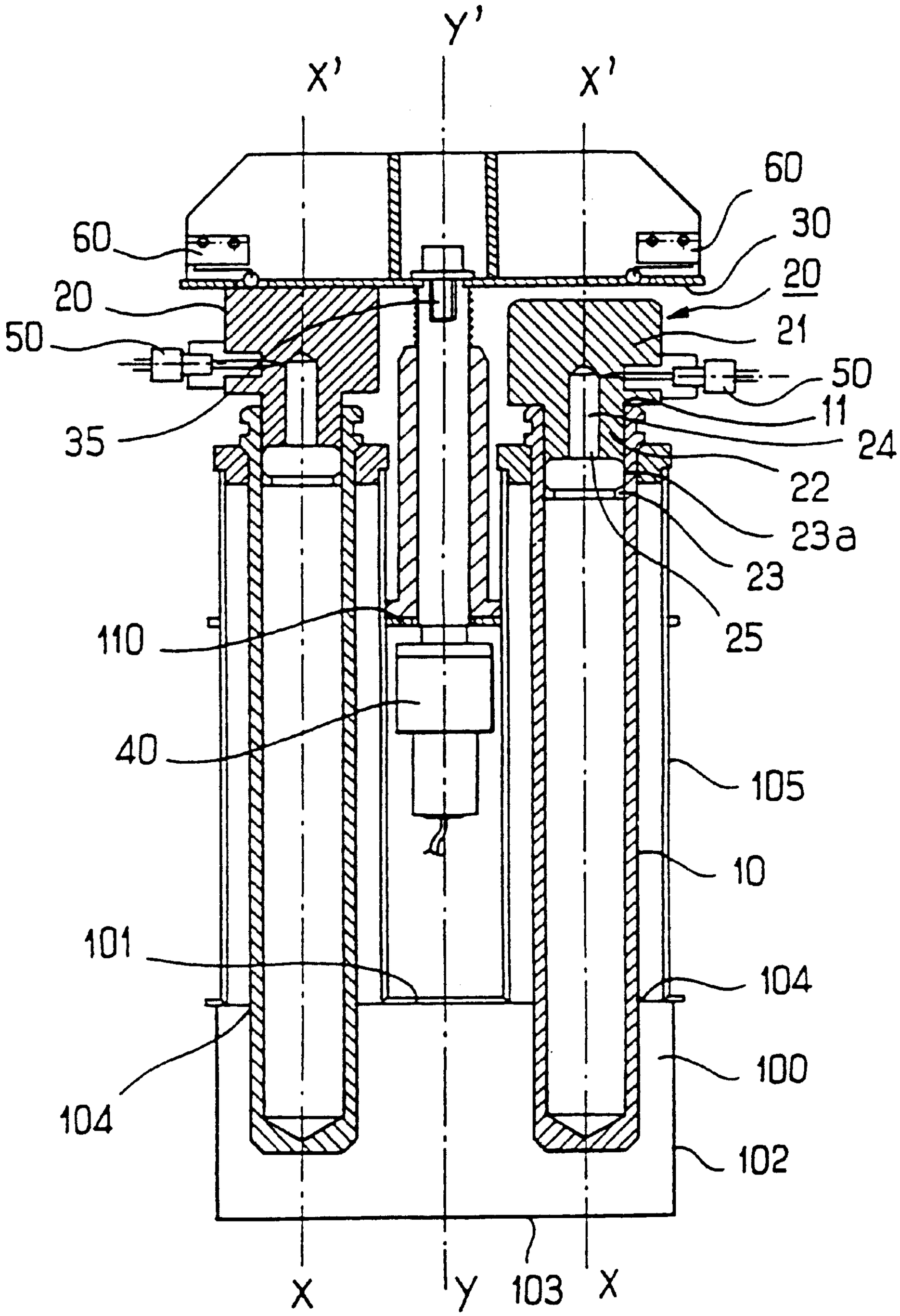

Y'
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FIG_1

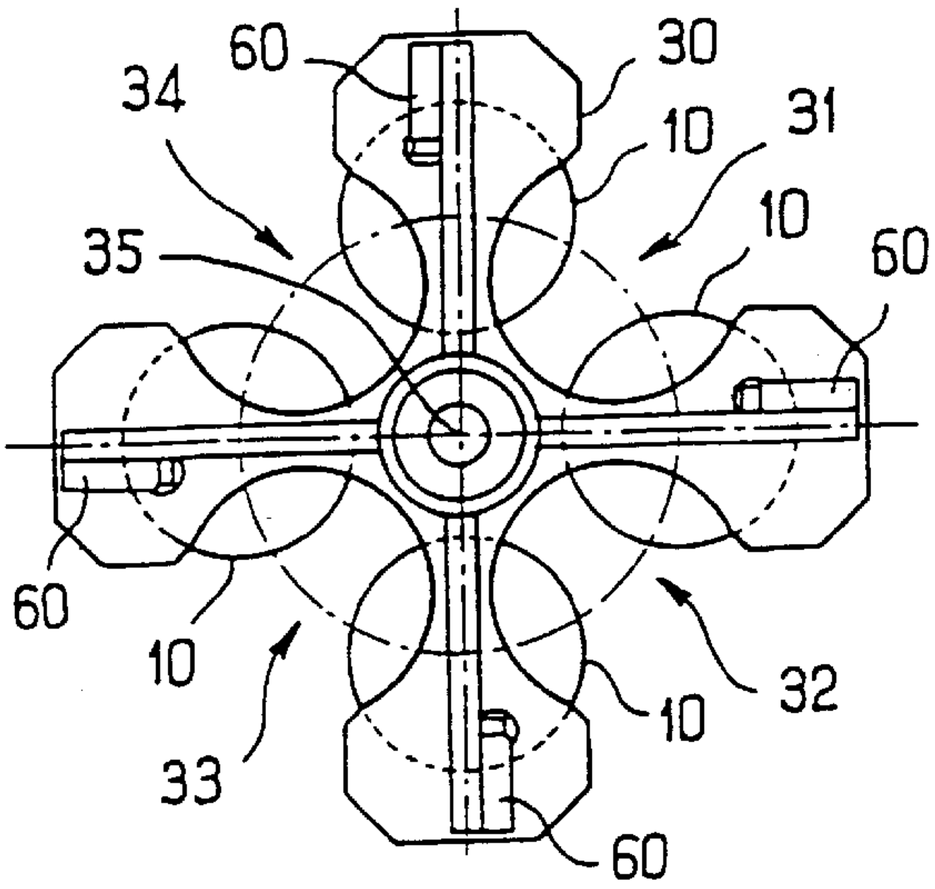

FIG_2

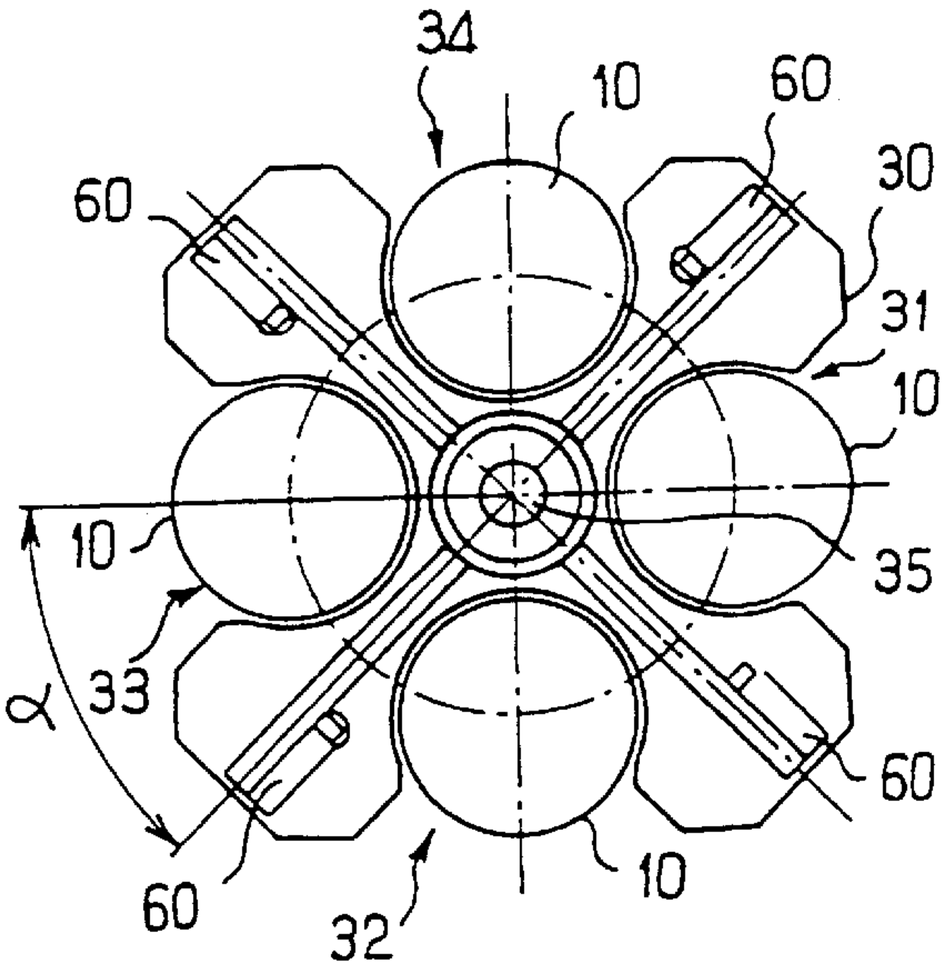

FIG_3

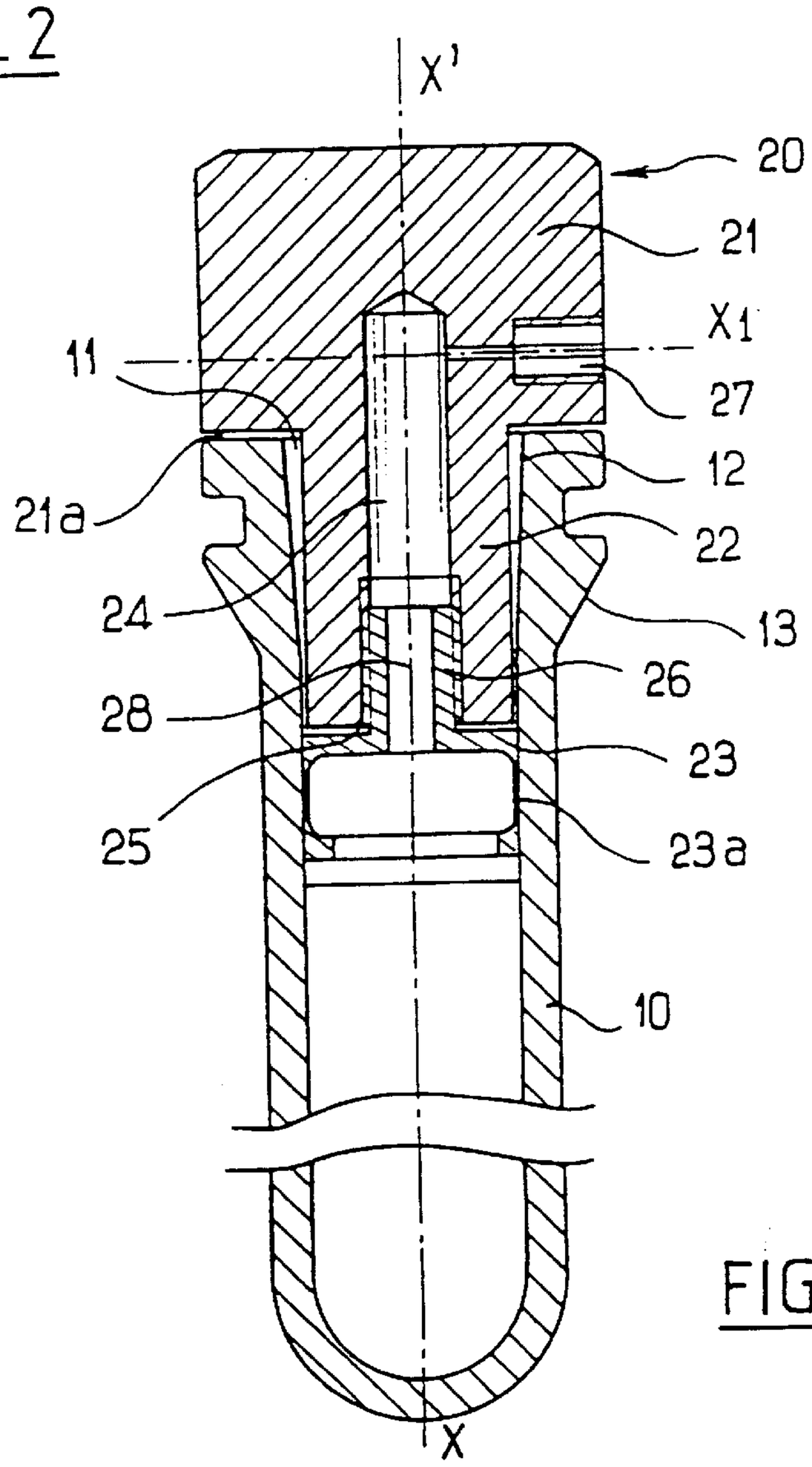

FIG_4

FLASK SEALING SYSTEM AND MICROWAVE TREATMENT APPARATUS WITH SUCH A SEALING SYSTEM

The present invention relates to a system for sealing at least one flask of longitudinal axis X–X' which is open at a first end for the introduction of a sample and which is positioned inside a microwave-application cavity with a view to heating the sample.

The invention also relates to apparatus for carrying out a treatment in a wet environment on a number of samples at the same time, the apparatus employing microwave heating of the samples and including such a sealing system.

Such apparatus finds a particularly advantageous use in carrying out chemical reactions such as mineralization, saponification, hydrolysis, speciation, and organic synthesis, for example.

Another particularly advantageous use of the apparatus according to the invention is the extraction under the effect of microwaves in a solvent of organic or inorganic compounds dispersed in soils, sediments, waters, plants, biological products or alternatively polymers.

In particular the invention relates to an improvement to the apparatus described in French patent applications nos 93 157 35 and FR 2 681 431 belonging to the Applicant Company, this apparatus including means for emitting microwaves into a microwave-application cavity of cylindrical shape operating in the open condition.

Already known apparatus of this type which operates in the closed condition, under pressure, generally comprises a microwave-application cavity in which bombs closed by screw-on caps are arranged.

This type of apparatus does, however, have several drawbacks.

This is because the bombs cannot contain large volumes of sample because the greater the volume of the sample, the more gas is given off under pressure when the sample is heated, which does not allow the bomb to work.

Furthermore, such apparatus working with bombs comprising screw-closure devices can work only in one condition, that is to say in the condition closed under pressure. It is not possible first of all to carry out degassing by making the apparatus work in the open condition then to close said apparatus to work under pressure.

The invention proposes a new sealing system which is fitted on a flask in a simple way allowing the flask to work in the open condition or closed condition, this making it possible to treat large volumes of samples by first of all carrying out an initial degassing operation before closing the flask.

More particularly, according to the invention, the sealing system includes:

a stopper intended to be pushed partially into said flask along the axis X–X' so as to seal its open end,

retaining means capable of holding said stopper in a position for sealing said associated flask, that part of the stopper which is engaged in said flask including a peripheral skirt which can be pressed in leaktight fashion against the internal wall of the flask under the effect of an overpressure which there is in this flask once the sample has been heated by microwaves.

According to a preferred embodiment of the invention, the means of retaining the sealing system comprise a bearing surface which can be positioned at right angles to the axis X–X' above each stopper, the relative position of the bearing surface and of the stopper allowing the latter to slide inside the flask along the axis X–X' in the direction of the bearing surface so as to come to bear against the latter under the thrusting force induced by the overpressure which there is inside the flask upon heating of the sample.

Furthermore, advantageously, in the sealing system in accordance with the invention, the skirt of each stopper is fitted with a lip capable of pressing in leaktight manner against the internal wall of the associated microwave-heated flask under the effect of the internal overpressure.

In accordance with the preferred embodiment of the sealing system according to the invention, it includes a pressure sensor connected to the bearing surface and capable of recording the overpressure which there is inside said flask through the bearing surface bearing on the stopper.

Furthermore, according to this preferred embodiment, the bearing surface of the sealing system in accordance with the invention carries at least one detector capable of coming into contact with each stopper bearing against said bearing surface and of emitting a signal which signals the presence of a stopper against said bearing surface.

According to a particularly advantageous feature of the sealing system in accordance with the invention, the bearing surface is mounted so that it can rotate about an axis Y–Y' parallel to the axis X–X' on a support secured to the application cavity between, on the one hand, an open position in which it is positioned offset from each flask giving unimpeded access to the open end of said flask for the fitting or the removal of the associated stopper or alternatively to allow said flask to be heated in the open condition, and, on the other hand, a closed position in which it is positioned above each flask fitted with the associated stopper, sealing its opening.

According to another embodiment of the sealing system in accordance with the invention, it includes a number of stoppers, each of said stoppers being intended to be pushed into a flask of axis X–X', said stoppers being capable of coming to bear against said bearing surface positioned at right angles to the axis X–X' above said stoppers under the thrusting force induced by the overpressure which there is inside said flasks upon microwave heating of the samples.

The description which will follow with reference to the appended drawing, which is given by way of non-limiting example, will make it easy to understand the essence of the invention and how it may be realized.

In the appended drawings:

FIG. **1** represents a diagrammatic view in longitudinal section of microwave-treatment apparatus including a sealing system in accordance with the invention,

FIG. **2** is a view from above of the apparatus of FIG. **1**, the sealing system being in a closed position above the flasks,

FIG. **3** is a diagrammatic view from above of the apparatus of FIG. **1**, the sealing system being in an open position,

FIG. **4** is a diagrammatic detail view of the stopper of the sealing system in accordance with the invention, the stopper being positioned inside a flask.

Represented in FIG. **1** is apparatus for carrying out a treatment in a wet environment on a number of samples which is contained in a number of flasks, here four flasks, the apparatus employing microwave heating of the samples.

Such apparatus is described structurally and functionally in its entirety in French patent applications FR 2 680 431 and no. 93 157 35 belonging to the Applicant Company. Consequently the apparatus as a whole will not be re-described here and the present description relates in detail only to the system for sealing such apparatus which corresponds to a specific embodiment thereof.

The application cavity **100** represented here is cylindrical of revolution of central axis of symmetry Y–Y' and includes

an upper wall **101**, a side wall **102** and a lower wall **103**. The side wall **102** of the application cavity **100** includes a window, not represented here, which is transparent to microwaves and via which a waveguide (also not represented) emerges inside said cavity. This waveguide is connected to a microwave generator, not represented, and conveys the microwaves as far as the application cavity. The upper wall **101** of the application cavity **100** here includes four circular openings **104**, only two of which are represented, these being arranged in twos symmetrically opposed with respect to the axis Y–Y'. Moreover, the application cavity **100** on the outside on its upper wall **101** carries four identical cylindrical wells **105**, two of which are represented here. Each cylindrical well **105** extends toward the outside of the cavity, at right angles to the upper wall **101**, starting from each circular opening **104** provided in said upper wall **101**. The section of each well **105** corresponds to the section of each opening **104** and the height of each well is such that it forms a microwave absorption barrier to avoid microwaves being propagated to the outside of the application cavity. The vertical axes X–X' of the cylindrical wells **105** are parallel to the axis Y–Y' and pass through the centers of the openings **104**.

The samples to be treated, not represented here, are placed in four flasks **10**, just two of which are represented in FIG. **1**. Each flask **10** is introduced vertically into each well **105** toward the application cavity **100**, via the openings **104** in such a way that part of the flask containing the sample lies in said cavity. When the flasks are positioned in the wells, the axes X–X' of the wells correspond to the axes X–X' of said flasks.

The flasks are made, for example, of quartz or of polytetrafluoroethylene.

Each flask **10** has an open upper end **11** positioned outside each well **105** and allowing the sample to be introduced into the flask **10**. As may be seen in greater detail in FIG. **4**, the outer surface **13** of each flask **10**, near its opening **11**, has a conical shape flared upward, which bears on the edge of the opening of each well **105**. Thus the edge of the opening of each well **105** forms a support for each flask positioned inside the application cavity **100**.

Furthermore, the apparatus represented in FIG. **1** includes a sealing system which comprises, for each flask **10**, a stopper **20** intended to be pushed partly into said flask along the axis X–X' so as to seal its open end **11**, and retaining means capable of retaining each stopper **20** in a position for sealing the associated flask. These retaining means consist of a closure lid comprising a bearing surface **30** capable of being positioned at right angles to the axis X–X' above each stopper **20**.

As can better be seen in FIG. **4**, each stopper **20** includes a head **21** which is cylindrical of revolution about the axis X–X' and protrudes outside each flask **10** when said stopper is partially pushed into the flask, and a body **22** extending substantially at right angles to said head **21** along the axis X–X', engaged with sliding along said axis X–X' in the flask **10**. The body **22** includes here at its free end **25** positioned inside the flask, a peripheral skirt **23** capable of pressing in leaktight fashion against the internal wall **12** of the associated flask under the effect of an overpressure which there is in this flask once a sample has been heated by microwaves. More specifically, this skirt **23** includes an annular lip **23***a* which preferably presses against the internal wall **12** of the flask **10** under the effect of the internal overpressure. It will be observed that the skirt **23** fitted with its lip **23***a* is cylindrical of revolution about the axis X–X', and is capable of interacting with a portion of the internal wall **12** of the flask which has an upwardly flared conical shape close to its opening **11**. This conical shape of the internal wall **12** of the flask advantageously allows the stopper **20** to be removed easily from the flask when the heating of the sample is over.

As can be seen in FIGS. **1** and **4**, each stopper **20** includes an axial duct **24** provided in the body **22** of each stopper which extends along the entire length of said body **22** as far as into the head **21**. This axial duct **24** which has symmetry of revolution about the axis X–X' is open at the free end **25** of the body **22** and is extended along the axis X–X' by the skirt **23** at this free end **25**. The axial duct **24** is connected, close to its other closed end situated in the head **21** of the stopper **20**, to a safety device **50** (represented diagrammatically in FIG. **1**). The safety device **50** and the axial duct **24** are connected together by means of a duct **27** of axis X_1 at right angles to the axis X–X' so that the safety device **50** is subjected to the overpressure which there is inside said flask through said axial duct **24** via the duct **27** of axis X_1. This safety device is capable of emitting a signal for shutting down the microwave heating above a given overpressure. The safety device **50** includes a blowout diaphragm, not represented here, positioned in such a way as to close off said duct **27** of axis X_1. This blowout diaphragm has the ability, for example, to withstand a pressure of approximately 40 bar, above which pressure it blows and sets off the emission of the shut-down signal.

It should be noted that according to the embodiment represented in FIG. **4**, the stopper **20** consists of two parts. A first part of the stopper **20** comprises the head **21** and the body **22** provided with the axial duct **24**, and a second part is formed by the peripheral skirt **23** which is extended at the top by a sleeve **26** intended to be fitted in the axial duct **24** along the axis X–X' in order to join the two parts together. Once assembled, the axis **28** of the sleeve **26** of the second part of the stopper is coincident with the axis X–X' of the axial duct **24**. Of course it is possible to envisage for the stopper **20** to be made as a single piece.

It should be emphasized that when there is no overpressure inside the flask **10**, then the lower surface **21***a* of the head **21** rests on the peripheral rim surrounding the opening **11** of the flask.

The sealing system in accordance with the invention and represented in FIGS. **1** to **3**, includes, as has already been explained, a lid including a bearing surface **30**. This lid is mounted so that it can rotate between a closed position and an open position (see FIGS. **2** and **3**) by means of a shaft **35**, about the axis Y–Y' of the application cavity, this axis Y–Y' being parallel to the axis X–X' of the flasks **10**, on a support **110** secured to the application cavity **100** and which extends transversely between the wells. This shaft **35** extends along the axis Y–Y' between the bearing surface **30** positioned at right angles above the stoppers **20** and a pressure sensor **40** positioned below the support **110** along the axis Y–Y'. The bearing surface **30** is mechanically connected to the pressure sensor **40**.

As can be seen more particularly in FIG. **1**, when there is no pressure inside said flasks **10**, the surface of the bearing surface **30** is positioned, when the lid is closed (see FIG. **2**) a certain distance away from the stoppers **20**. The relative position of the bearing surface **30** and of the stoppers **20** allows the stoppers to slide inside the flasks along the axis X–X' in the direction of the bearing surface **30** so that they come to bear against the latter (see FIG. **1**) under the thrusting force induced by the overpressure which there is inside the flasks **10** upon heating of the samples. In this position in which each stopper **20** is bearing, the peripheral skirt **23** equipped with its lip **23***a* presses in leaktight manner

against the internal wall **12** of the associated flask. It will be noted that for this the thrusting force induced by the overpressure which there is in each of said flasks is greater than the gripping force of the peripheral skirt **23** against the internal wall **12** of each flask so that it is possible to open the flask by positioning the lid in the open position (see FIG. **3**) once heating is over.

As can also be seen in FIG. **1**, the bearing surface **30** carries detectors **60** which are capable of coming into contact with the stoppers **20** bearing against said bearing surface **30** and intended to emit a signal which signals the presence of each stopper **20** against the bearing surface **30**. Thus advantageously it is possible to check that each flask is closed using these detectors, checking the presence of the stopper **20** bearing against the bearing surface **30**.

As may be seen more particularly in FIGS. **2** and **3**, the lid and the bearing surface **30** have a special shape including cells **31**, **32**, **33**, **34** of essentially circular shape. According to the embodiment represented, the bearing surface **30** has four cells. Each of the circular cells has a diameter slightly larger than the diameter of a flask **10**. The cells **31**, **32**, **33**, **34** are positioned in twos symmetrically with respect to the axis Y–Y'.

When the lid is in the open position (represented more particularly in FIG. **3**), each flask **10** is positioned in each cell **31**, **32**, **33**, **34** of the bearing surface **30** in such a way that said bearing surface **30** is positioned offset from each of the flasks **10**. Thus there is unimpeded access to the upper opening **11** of each flask **10** for the fitting or removal of a stopper **20** or alternatively to allow the microwave apparatus and associated flasks to operate open. The latter case may be envisaged for initial degassing of the flasks at the beginning of heating.

When the lid is in the closed position (see FIG. **2**), the bearing surface **30** is positioned in a position offset by an angle α from the open position of the lid. In this closed position, the bearing surface **30** is placed above each stopper **20** pushed into each flask **10**. Thus as the pressure inside the flasks **10** rises, each stopper **20** comes to bear against said bearing surface **30** and provides a seal at the opening **11** of each flask **10**.

As has already been explained, the bearing surface **30** is connected under the support **110** to a pressure sensor **40**. This pressure sensor **40** is capable of recording the overpressure which there is in all of the four flasks through the bearing surface bearing on the stoppers **20**. This pressure sensor works in tension. This is because the stoppers **20** bearing on the bearing surface **30** exert on the bearing surface **30** an upward tensile force which leads to the compression of a liquid or of calibrated crystals contained within the pressure sensor which allows the sum of the pressures in the flasks to be measured. It is thus possible to evaluate a mean value of the pressure in said flasks. Such a sensor is set to approximately 10 to 15 bar. This sensor advantageously makes it possible to check and control the pressure inside the flasks **10** and may allow automatic control of the apparatus on the basis of the pressure measured.

The present invention is not in any way limited to the embodiment described and represented but those skilled in the art will be able to envisage any alternative forms which conform to its spirit.

Indeed, it is possible to envisage apparatus comprising a greater number of flasks (6, 8 or 10 flasks) comprising a sealing system according to the invention.

I claim:

1. System for sealing at least one flask **(10)** of longitudinal axis X–X', which is open at a first end **(11)** intended to allow the introduction of a sample and which is positioned inside a microwave-application cavity **(100)** with a view to heating the sample, characterized in that it includes:

a stopper **(20)**, intended to be pushed partially into said flask **(10)**, which is movable along the axis X–X' so as to seal the open end **(11)**, retaining means, having bearing surface **(30)**, which is mounted securely on a support **(110)** forming an integral part of the application cavity **(100)** in order to hold said stopper **(20)** in a position for sealing the associated flask **(10)**, the retaining means comprising a bearing surface **(30)** which can be positioned at right angles to the axis X–X' above each stopper **(20)**, the relative position of the bearing surface **(30)** and of the stopper **(20)** allowing the latter to slide inside said flask **(10)** along the axis X–X' in the direction of said bearing surface **(30)** so as to come to bear against the latter under the thrusting force induced by the overpressure which there is inside said flask **(10)** upon heating of the sample,

part **(22)** of the stopper **(20)** which is engaged in said flask **(10)** including a peripheral skirt **(23)** which can be pressed in leaktight fashion against the internal wall **(12)** of the flask **(10)** under the effect of an overpressure which there is in the flask once the sample has been heated by microwave.

2. Sealing system according to claim **1**, characterized in that the peripheral skirt **(23)** of each stopper **(20)** has an external wall which is cylindrical of revolution about the axis X–X' and capable of pressing against said substantially conical internal wall **(12)** of said flask **(10)**.

3. Sealing system according to claim **2**, characterized in that it includes a pressure sensor **(40)** connected to the bearing surface **(30)** and capable of recording the overpressure which there is inside said flask **(10)** through said bearing surface **(30)** bearing on said stopper **(20)**.

4. Sealing system according to claim **2**, characterized in that the bearing surface **(30)** carries at least one detector **(60)** capable of coming into contact with each stopper **(20)** bearing against said bearing surface **(30)** and of emitting a signal which signals the presence of said stopper **(20)** against said bearing surface **(30)**.

5. Sealing system according to claim **2**, characterized in that the bearing surface **(30)** is mounted so that it can rotate about an axis Y–Y' parallel to the axis X–X' on said support **(110)** secured to the application cavity **(100)** between, on the one hand, an open position in which it is positioned offset from each flask **(10)** giving unimpeded access to the open end of said flask **(10)** for the fitting or the removal of the associated stopper **(20)** or alternatively to allow said flask **(10)** to be heated in the open condition, and, on the other hand, a closed position in which it is positioned above each flask **(10)** fitted with the associated stopper **(20)**, sealing its opening **(11)**.

6. Sealing system according to claim **2**, characterized in that it includes a number of stoppers **(20)**, each of said stoppers **(20)** being intended to be pushed into a flask **(10)** of axis (X–X'), said stoppers **(20)** being capable of coming to bear against said bearing surface **(30)** positioned at right angles to the axis X–X' above said stoppers **(10)** under the thrusting force induced by the overpressure which there is inside said flasks **(10)** upon microwave heating of the samples.

7. Sealing system according to claim **1**, characterized in that the peripheral skirt **(23)** of each stopper **(20)** is fitted with a lip **(23*a*)** capable of pressing in leaktight manner against the internal wall **(12)** of the associated microwave-heated flask **(10)** under the effect of the internal overpressure.

8. Sealing system according to claim 1, characterized in that each stopper (20) has a head (21) which protrudes outside each flask (10) and a body (22) which extends substantially at right angles to said head (21), engaged with sliding along the axis X–X' inside said flask (10) and being provided with an axial duct (24) which is open at the free end (25) of said body (22), said skirt (23) being positioned level with said free end (25) in the axial extension of said duct (24).

9. Sealing system according to claim 8, characterized in that the axial duct (24) extends from the free end (25) of said body (22), through the entire length of said body (22), and into said head (21) to where it is connected to a safety device (50) intended to be subjected to the overpressure which there is inside said flask (10) by means of said axial duct (24) and capable of emitting a signal for shutting down the microwave heating above a given overpressure.

10. Apparatus for carrying out a treatment in a wet environment on a number of samples which are contained in a number of flasks (10) of axis X–X', the apparatus comprising means for emitting microwaves into an application cavity (100) of central axis of symmetry Y–Y', the application cavity (100) in its upper wall (101) including openings for the introduction of the flasks (10) inside the application cavity (100) parallel to the axis Y–Y', these being arranged in twos so that they are symmetrically opposed with respect to the axis Y–Y', characterized in that it includes a sealing system according to claim 1.

11. System for sealing at least one flask (10) of longitudinal axis X–X', which is open at a first end (11) intended to allow the introduction of a sample and which is positioned inside a microwave-application cavity (100) with a view to heating the sample, characterized in that it includes:

a stopper (20), intended to be pushed partially into said flask (10) along the axis X–X' so as to seal its open end (11),

retaining means, having bearing surface (30), which is mounted securely on a support (110) forming an integral part of the application cavity (100) in order to hold said stopper (20) in a position for sealing the associated flask (10),

part (22) of the stopper (20) which is engaged in said flask (10) including a peripheral skirt (23) which can be pressed in leaktight fashion against the internal wall (12) of the flask (10) under the effect of an overpressure which there is in this flask once the sample has been heated by microwaves,

characterized in that each stopper (20) has a head (21) which protrudes outside each flask (10) and a body (22) which extends substantially at right angles to said head (21), engaged with sliding along the axis X–X' inside said flask (10) and being provided with an axial duct (24) which is open at the free end (25) of said body (22), said skirt (23) being positioned level with said free end (25) in the axial extension of said duct (24), and characterized in that the axial duct (24) extends from the free end (25) of said body (22), through the entire length of said body (22), and into said head (21) to where it is connected to a safety device (50) intended to be subjected to the overpressure which there is inside said flask (10) by means of said axial duct (24) and capable of emitting a signal for shutting down the microwave heating above a given overpressure.

12. Apparatus for carrying out a treatment in a wet environment on a number of samples which are contained in a number of flasks (10) of axis X–X', the apparatus comprising means for emitting microwaves into an application cavity (100) in its upper wall (101) including openings for the introduction of the flasks (10) inside the application cavity (100) parallel to the axis Y–Y', these being arranged in twos so that they are symmetrically opposed with respect to the axis Y–Y', characterized in that it includes a system for sealing said flasks (10) of longitudinal axis X–X', which is open at a first end (11) intended to allow the introduction of a sample and which is positioned inside a microwave-application cavity (100) with a view to heating the sample, characterized in that it includes:

a number of stoppers (20) intended to be pushed partially into said flasks (10) along the axis X–X' so as to seal its open end (11),

retaining means, having bearing surface (30), which is mounted securely on a support (110) forming an integral part of the application cavity (100) in order to hold said stoppers (20) in a position for sealing the associated flasks (10),

part (22) of each stopper (20) which is engaged in each flask (10) including a peripheral skirt (23) which can be pressed in leaktight fashion against the internal wall (12) of each flask (10) under the effect of an overpressure which there is in each flask once the sample has been heated by microwaves.

13. Apparatus for carrying out a treatment in a wet environment on a number of samples which are contained in a number of flasks (10) of axis X–X', the apparatus comprising means for emitting microwaves into an application cavity (100) in its upper wall (101) including openings for the introduction of the flasks (10) inside the application cavity (100) parallel to the axis Y–Y', these being arranged in twos so that they are symmetrically opposed with respect to the axis Y–Y', characterized in that it includes a system for sealing said flasks (10) of longitudinal axis X–X', which is open at a first end (11) intended to allow the introduction of a sample and which is positioned inside a microwave-application cavity (100) with a view to heating the sample, characterized in that it includes:

a number of stoppers (20) intended to be pushed partially into said flasks (10) along the axis X–X' so as to seal it open end (11),

retaining means, which is mounted securely on a support (110) forming an integral part of the application cavity (100) in order to hold said stoppers (20) in a position for sealing the associated flasks (10), the retaining means comprising a bearing surface (30) which can be positioned at right angles to the axis X–X' above the stoppers (20), the relative position of the bearing surface and of the stoppers allowing the latter to slide inside the flasks (10) along the axis X–X' in the direction of the bearing surface so as to come to bear against the latter under the thrusting force, part (22) of each stopper (20) which is engaged in each flask (10) including a peripheral skirt (23) which can be pressed in leaktight fashion against the internal wall (12) of each flask (10) under the effect of an overpressure which there is in each flask once the sample has been heated by microwaves induced by the overpressure which there is inside said flasks (10) upon heating of the sample.

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